AGP Lab Blank Analysis and Blank Data Review

1. SCOPE AND APPLICATION

1.1. This method was developed by laboratory management as part of the quality assurance protocol for Algal Growth Potential (AGP) testing. It is applicable to both freshwater and saltwater AGP tests.

2. SUMMARY OF METHOD

2.1. This SOP outlines the procedure for analyzing laboratory method blanks to monitor contamination of a typical sample. Examples of such contamination are nutrients carried over in the inoculum, unwanted materials in DI rinse water, and foreign particles from improperly cleaned sample bottles. By definition, these blanks are not to be treated differently from other samples in terms of preparation, set-up, and counting. This method also describes the review and corrective action for data from lab blanks.

2.2. Interferences:

2.2.1. Contaminated glassware

2.2.2. Airborne particulates including dust and microscopic organisms

3. EQUIPMENT AND SUPPLIES

3.1. General

3.1.1. Mettler Toledo SevenMulti® meter (SN 1225117049) (Calibrated per SOP TA-06.25)

3.1.2. Accumet Model AR 10 pH meter (SN AR 93312105) (Calibrated per SOP TA-06.23)

3.1.3. Labeling tape (purple or orange colored tape, for freshwater and saltwater blanks respectively, is generally used to indicate blank containers but any color that differentiates from an actual sample may be used)

3.1.4. Specimen cup, 30 mL, one per blank

3.1.5. Calibrated thermometer (located in B246 thermometer drawer) or certified digital thermometer (located in B246 – white box to left of telephone) See SOP TA-06.13 and LB-035.

3.1.6. *AGP Sample Log-in Record* logbook

3.2. For freshwater

3.2.1. AGP sample container

3.2.2. Deionized (DI) water

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3.2.2.1. Distilled water can be substituted for DI water at the analyst's discretion (ex. problems with DI water system). If distilled water is used, substitute distilled water for DI water throughout the SOP.

3.3. For saltwater

3.3.1. Erlenmeyer flask

3.3.2. Autoclaved DI water (autoclave SOP TA-06.18)

3.3.3. Mettler PB403-S/Fact balance (SN 1123052001) (Calibrated per SOP TA-06.31)

3.3.4. Small weigh boat (plastic if available)

3.3.5. Metal spatula

3.3.6. Magnetic stir plate

3.3.7. Magnetic stir bar, 2.5 cm

3.3.8. Stir bar retriever

3.3.9. Crystal Sea® Marine Mix Bioassay Laboratory Grade artificial sea salts

4. METHODS

4.1. For freshwater blank

4.1.1. Label a clean AGP bottle using purple label tape. Write "Lab Blank," date collected and analyst's initials on the label.

4.1.2. Fill the bottle with DI water from the sink.

4.1.3. Enter "Lab Blank" in the *AGP Sample Log-in Record* under the LIMS # column.

4.1.3.1. A log book label can be made per SOP TA-08.09.

4.1.4. Measure conductivity, and salinity with the Mettler Toledo SevenMulti® meter. Measure temperature with the certified/calibrated digital thermometer. Measure pH with the Accumet AR 10 pH meter. Record the values in the appropriate columns of the *AGP Sample Log-in Record*.

4.1.4.1. See SOP TA-06.23 for measuring pH and SOP TA-06.25 for measuring conductivity and salinity.

4.1.5. Autoclave and filter the blank.

4.1.5.1. Autoclave and filter per SOP TA-08.01 for samples of <1.0 ppth salinity.

4.1.6. If the blank is to be set up immediately, leave it out. Otherwise, place the blank in the walk-in cooler.

4.1.6.1. Set up blanks per SOP TA-08.02.

4.2. For saltwater blank

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4.2.1. Label a clean 500-mL Erlenmeyer flask using orange label tape. Write "Salt Blank," date collected and analyst's initials on the label.

4.2.2. Fill the flask approximately half full with autoclaved DI water.

4.2.3. Scoop 3-4 grams of artificial sea salts into a weigh boat using the metal spatula. Use balance for weighing if desired.

4.2.4. Place the flask on the magnetic stirrer. Place the stir bar in the flask and turn the stirrer on.

4.2.5. Add a small amount of artificial sea salts to the flask using the metal spatula and wait until the salts are dissolved.

4.2.6. Measure the salinity using the SevenMulti® meter. If the salinity is less than 6.9 ppth (parts per thousand), repeat STEP 4.2.5. above until the salinity measures 7±0.1 ppth. If the salinity of 7±0.1 ppth is exceeded, dilute with autoclaved DI water. Repeat salinity measurement as needed.

4.2.6.1. See SOP TA-06.25 for measuring salinity.

4.2.6.2. If dilution is needed, gradually add DI water to the mixture. Swirl to mix, then repeat the salinity measurement.

4.2.7. Remove the flask from the stirrer and turn the stirrer off. Use the stir bar retriever to remove the stir bar from the flask.

4.2.8. Enter "Salt Blank" in the *AGP Sample Log-in Record* under the LIMS # column.

4.2.8.1. A log book label can be made per SOP TA-08.09.

4.2.9. Measure conductivity, and salinity with the Mettler Toledo SevenMulti® meter. Measure temperature with the certified/calibrated digital thermometer. Measure pH with the Accumet AR 10 pH meter. Record the values in the appropriate columns of the *AGP Sample Log-in Record*.

4.2.9.1. See SOP TA-06.23 for measuring pH and SOP TA-06.25 for measuring conductivity. Record the salinity as measured in Step 4.2.6.

4.2.10. Filter the blank.

4.2.10.1. Filter per SOP TA-08.01 using a 0.45 μm filter.

4.2.11. If the blank is to be set up immediately, leave it out. Otherwise, place the blank in the walk-in cooler.

4.2.11.1. Set up blanks per SOP TA-08.02.

5. DATA REVIEW

5.1. Select lab blank results during the AGP result selection process.

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5.1.1. Results are selected per SOP TA-08.07. This should be done before each run's Excel file is printed for result verification in case the data must be qualified.

5.2. After the data for each run has been checked by two-person verification, open the folder X:\Labs\Lab5\Biology\Bench Common\ToxCommon\AGP\Control Charts.

5.2.1. Data-Entry Verification per SOP BG-01.

5.3. Open Excel spreadsheet named **Lab Blanks**.

5.4. Select either the **FW Table** tab for freshwater blanks or **SW Table** tab for saltwater blanks, scroll down to the first available line, and enter the lab blank's set-up date under **Analysis Date** header.

5.4.1. If folder is locked please see supervisor.

5.5. Enter the lab blank values for days 10, 12, and 14 in the next three columns. When a value is not available for a day, enter NV (no value).

5.6. In the column titled Reported Value, enter in the result that was selected.

5.6.1. Results are selected per SOP TA-08.07.

5.7. Fill each remaining cell in the row with the formula from the cell above it.

5.7.1. See Microsoft Excel Help for help with table functions.

5.8. Click the **FW Chart- Year** tab or **SW Chart- Year** tab and update the source data of the chart to reflect the newly entered data. This chart should display the lab blank maximum values for the past 12 months.

5.9. Click the **FW Chart- Last 20** tab or **SW Chart- Last 20** and update the source data of the chart to reflect the newly entered data. This chart should reflect the 20 most recent lab blank maximum values.

5.10. If the lab blank's maximum value exceeds the MDL (0.300 for freshwater and 3.00 for saltwater), then all samples from that batch should be qualified with a "V" qualifier, which indicates that the analyte was detected in both the blank and the sample. Qualify sample data where necessary and notify a supervisor of the problem.

5.11. If a blank exceeds the control limit or if 2 of 3 successive blanks exceed the warning limit, the samples should be set up again in the next run (where sufficient sample is available). Notify a supervisor of the problem.

5.11.1. See supervisor if the blanks exceed the MDL and the control limit or if 2 of 3 successive blanks exceed the warning limit.

5.11.2. The value of the current Mean+1SD of the 20 most recent blanks is the warning limit. The value of the current Mean+2SD is the control limit.

5.11.3. If the subsequent lab blank also exceeds the limit in question, analyses should stop while possible causes of contamination and error in method are examined.

5.12. Save the file and exit, but see supervisor first.

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6. DATA ARCHIVAL

6.1. Use a black, waterproof pen for all bench sheets and logbooks.

6.2. Record information in *AGP Sample Log-in Record* logbook.

6.3. To correct raw data entries, place a single line through the incorrect entry, write the corrected entry near the error, date and initial the correction. Do not overwrite entries.

6.3.1. The corrected entry is written as near its appropriate space as possible or else in the comment box on the appropriate bench sheet.

6.3.2. If the correction requires an explanation, write a comment and reference the error. Initial and date the explanation.

6.4. Use sequential alphabetical annotation to identify errors and corresponding comments (use A for first error and use A for corresponding explanatory comment; use B for second error, etc.).

7. QUALITY CONTROL

7.1. Refer to QA manual for corrective action and handling out-of-control data.

8. SAFETY/HAZARDOUS WASTE MANAGEMENT

8.1. Proper laboratory personal protection, including lab hood or ventilation system, lab coat, gloves and safety glasses, is required when working with chemicals and unprocessed samples.

8.2. Refer to Laboratory Quality Manual for sample disposal

8.3. Refer to the Laboratory Health and Safety Manual and Hazardous Waste Management

9. REFERENCES

9.1. Laboratory Quality Manual http://www.dep.state.fl.us/labs/library/lab_sops.htm

9.2. Laboratory Health and Safety Manual and Hazardous Waste Management https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents

9.3. DEP SOP TA-08.01, Initial Algal Assay Sample Handling

9.4. DEP SOP TA-08.02, Algal Growth Potential (AGP) and Limiting Nutrient (LN) Algal Assay Set-Up

9.5. DEP SOP TA-08.07, Algal Growth Potential and Limiting Nutrient Data Verification and LIMS Upload

9.6. DEP SOP TA-08.09, Sample Custody, Log Book Labels and Prep Worksheet Manager for Algal Growth Potential (AGP) Samples

9.7. DEP SOP TA-06.31, Calibration and Use of Mettler PB403-S/FACT Balance

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9.8. DEP SOP TA-06.23, Calibration and Use of Accumet Model AR10 pH Meter

9.9. DEP SOP TA-06.25, Calibration and Use of Mettler Toledo SevenMulti® for Measuring Conductivity, Salinity and TDS

9.10. DEP SOP BG-01, Data-Entry Verification

APPENDIX OF CHANGES

12/20/2007 Added Scope and Application; Summary of Method, and Appendix of Changes.

10/08/2013 Reformatted SOP.

09/11/2014 Changed temperature measurement to digital thermometer.

01/28/2015 Formatted for 508 compliance.

08/17/2015 Updated links. Added instrument Serial Numbers.

09/15/2016 Updated links and references.

09/14/2017 Updated the Control Chart file location in the Data Review section.

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